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SIMULATIONS OF CREEP IN DUCTILE-PHASE TOUGHENED Nb₅Si₃/Nb *IN-SITU* COMPOSITES

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ABSTRACT

The primary and steady-state creep behavior of ductile-phase toughened Nb₅Si₃/Nb *in-situ* composites has been simulated using analytical and finite element (FE) continuum techniques. The microstructure of these composites is complex, consisting of large, elongated primary dendrites of the ductile (Nb) solid-solution phase in a eutectoid matrix with the silicide as the continuous phase. This microstructure has been idealized to facilitate the modeling; the effects of these idealizations on the predicted composite creep rates are discussed. Further, it has been assumed that the intrinsic creep behavior of each phase within the composite is the same as that of the corresponding bulk material. Thus, the experimentally measured creep properties of the bulk Nb₅Si₃ and (Nb) phases have been analyzed to provide the required material constants in the creep constitutive equation. Model predictions of the steady-state composite creep rate have been compared with the experimental results for a Nb-10 at.% Si alloy. While accurate at low stress, the models underpredict the composite creep rate at large stresses because the composite stress exponent is underpredicted. In the case of primary creep, the models somewhat overpredict the composite creep strain but are reasonably accurate given uncertainties in the primary creep data. Finally, FE predictions of the tensile stress distributions within the composites have been shown to be qualitatively consistent with the cracking observed experimentally during tertiary creep.

INTRODUCTION

The favorable fracture behavior of ductile-phase toughened (DPT), *in-situ* composites of Nb and Nb₅Si₃ has been demonstrated recently by several research groups, *e.g.* [1-3]. The oxidation resistance of alloys based on this system also appears promising [4], suggesting that Nb₅Si₃/Nb composites have potential as advanced structural materials for high temperature service. However, the high temperature creep behavior of these composites has not been reported to date. In a companion paper to this article [5], the creep behavior of a Nb-10 at.% Si *in-situ* composite was experimentally determined. In addition, the creep properties were determined for the bulk component phases: Nb₅Si₃ and a Nb-1.25 at.% Si solid solution, hereafter denoted (Nb). The results of that experimental study, which are summarized below, were used to perform the numerical simulations of primary and steady-state creep reported here.

The simulations presented in this paper make use of continuum methods. Following earlier work on ideal DPT composites [6,7], both a finite element (FE) and a simple analytical approach were taken. The continuum approach has been fairly successful in modeling deformation of metal matrix composites (MMCs) [8-10], and was judged to be a reasonable

starting point for simulating creep in DPT Nb₅Si₃/Nb composites. It is important to note, however, that there are two features of deformation in DPT composites, such as Nb₅Si₃/Nb, that separate them from the well-studied case of MMCs: (1) both phases undergo time-dependent deformation, and (2) the matrix (*i.e.* the continuous phase) is the stronger, or more creep resistant, phase [5]. Thus, the major purpose of the work described here was to determine whether the continuum approach is useful in modeling creep of DPT composites such as Nb₅Si₃/Nb.

SUMMARY OF THE EXPERIMENTS

As described in more detail by Subramanian *et al.* [5], Nb₅Si₃/Nb composites with a bulk composition of Nb-10 at.% Si were prepared by casting, extruding at 1482 °C to a 10:1 ratio, and annealing at 1500 °C for 100 h. The resulting microstructure consisted of large (~50 µm) elongated proeutectic (Nb) dendrites in a eutectoid matrix composed of small (~5 µm) (Nb) particles dispersed within the continuous Nb₅Si₃ phase. From the phase diagram, the total (Nb) volume fraction is expected to be approximately 73%. In addition, pure Nb₅Si₃ was prepared by arc melting, crushing to a powder, and vacuum hot pressing at 34.5 MPa and 1700-1800 °C for 4 h. This process was followed by annealing at 1500 °C for 100 h. The ductile (Nb) constituent in the composites, a solid solution of Nb-1.25 at.% Si, was prepared in bulk form by casting, extruding to a 12:1 ratio at 1482 °C, and annealing at 1500 °C for 16 h.

Creep testing of the composites and the bulk component phases was performed in a high purity flowing argon atmosphere using a constant-load lever-arm creep frame. The composites and the (Nb) were tested in tension, while the brittle Nb₅Si₃ was tested in compression. SLVC extensometers with a 5 micro-strain resolution were used to measure specimen displacements for all tests (both tension and compression), thereby avoiding errors due to load train compliance. Both constant load and load-increase tests were performed at temperatures from 1100 to 1350 °C, though only 1200 °C tests were numerically simulated. The results of these experiments are described in the companion paper [5]. The key results from the standpoint of the numerical modeling are given in the following sections.

NUMERICAL METHODS

As described in detail elsewhere [6,7], the FE simulations were performed using the “unit cell” approach [8-10]. In this approach, the composites were assumed to consist of a regular array of discontinuous cylindrical fibers embedded in a continuous matrix. The two-dimensional axisymmetric boundary value problem was solved for one unit cell comprising one fiber embedded within a cylinder of the surrounding matrix in proportions to match the volume fractions actually present in the idealized composite. Perfect bonding between the two phases and periodic boundary conditions were assumed. The FE calculations were performed using the NIKE2D solid mechanics code [11]. A constant applied axial stress was simulated by continuously decreasing the applied axial force during the simulation to account for the reduction in the radius of the deformed mesh. In addition to elastic deformation, both of the phases in the composite were assumed to creep according to the equation [12]:

$$\varepsilon_{cr} = A (\sigma_{eff})^n t^m, \quad (1)$$

where ε_{cr} is the von Mises effective creep strain, σ_{eff} is the von Mises effective stress, t is time, n is the stress exponent, and A and m are constants. Primary creep is simulated by a time exponent, m , less than unity. For the case of $m = 1$, or steady-state creep, the strain rate reduces to the familiar Norton power law.

For comparison with the FE analysis, a simple analytical model of composite creep based on the work of Bullock *et al.* [13] and Tanaka *et al.* [14] was developed [7]. This model assumes strain compatibility between the phases and uniform stress and strain states within each phase (the FE approach does not require these assumptions). The constitutive behavior of each phase in this model is given by adding the creep strain, eq. (1), to the elastic strain. As described previously [7], by adding a rule-of-mixtures constraint on the stress in each phase, a simple set of equations can be derived and integrated to give the stress in each phase and the total composite strain as a function of time.

A preliminary Nb₅Si₃/Nb composite model was employed in the FE calculations of Henshall and Strum [7], in which the matrix was assumed to be pure Nb₅Si₃. The equilibrium volume fraction of 73% (Nb) was assumed to be contained completely within the primary dendrites, which were approximated as cylindrical rods with a 5:1 aspect ratio. In the present study, linear intercept analyses of the actual Nb-10% Si composite microstructure led to a refinement in the microstructural idealization. First, the matrix was simulated as a eutectoid microconstituent consisting of 36% equiaxed (Nb) particles in a continuous Nb₅Si₃ phase. Within this eutectoid matrix, the primary (Nb) dendrites were approximated as cylindrical rods with an 8:1 aspect ratio and a 64% volume fraction.

In the preliminary study only steady-state creep was considered and (Nb) creep properties were estimated from data for pure Nb and a Nb-4% Si alloy [7]. In the present study both primary and steady-state creep were considered, and actual (Nb) and Nb₅Si₃ data from Subramanian *et al.* [5] were used to evaluate the material constants in eq. (1). The derivation of these values employed the standard regression techniques. One complication, however, was that the FE code could not integrate eq. (1) for $n < 1$. For pure Nb₅Si₃, the steady-state value of n was determined to be about 0.85 but a value of $n = 1.0$ was used in the calculations to avoid this numerical problem. Figure 1 demonstrates that this approximation provides a reasonable fit to the data. Further, the material constants for the eutectoid microconstituent were estimated by performing analytical model calculations for a composite containing 36% (Nb) particles in an Nb₅Si₃ matrix using the material constants determined for (Nb) and pure Nb₅Si₃. Primary creep constants were determined only for the pure Nb₅Si₃ and (Nb). Nb₅Si₃ data were available only at one stress. In this case, the model fit to the data was essentially perfect, indicating that for a single stress eq. (1) accurately represents primary creep in the silicide. Data at three stresses were available for the (Nb) so compromises were necessary in selecting the constants, resulting in a less accurate fit for any single stress, Fig. 2. Thus, eq. (1) is less than ideal in representing primary creep of the solid solution alloy. However, the steady-state results presented in the next section suggest that the computed composite creep strain may be fairly insensitive to variations

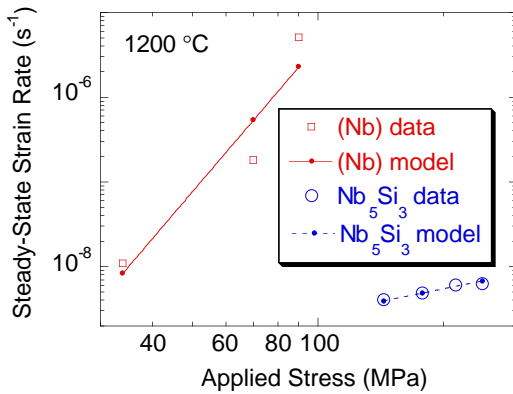
in the (Nb) behavior. All of the material constants employed in the numerical predictions are given in Table I.

Table I. Material Constants Used in the Creep Predictions.

Case	Microconstituent	E (GPa)	ν	A (MPa $^{-n} \cdot \text{s}^{-1}$)	n	m
Steady state (Preliminary)	Nb ₅ Si ₃	121 (327)	0.3 (0.38)	2.728x10 ⁻¹¹ (3.63x10 ⁻¹¹)	1.0 (1.0)	1.0 (1.0)
“	(Nb)	72 (103)	0.3 (0.38)	1.235x10 ⁻¹⁷ (2.98x10 ⁻¹⁶)	5.76 4.68	1.0 (1.0)
“	Eutectoid	121	0.3	2.015x10 ⁻¹¹	1.129	1.0
Primary	Nb ₅ Si ₃	121	0.3	1.790x10 ⁻⁵	1.0	0.1171
“	(Nb)	72	0.3	1.496x10 ⁻⁹	2.64	0.4617

Note: E is the Young's modulus and ν is Poisson's ratio.

(1)



(2)

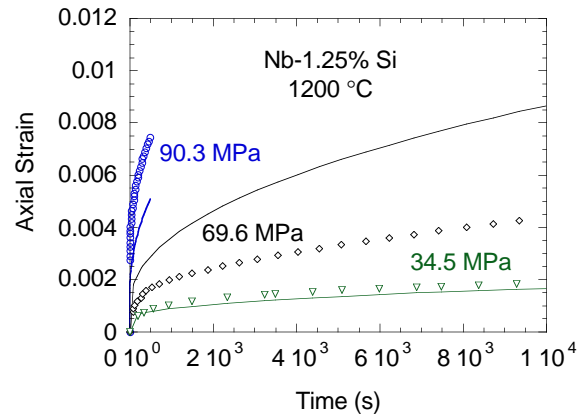


Figure 1. Comparison of the data [5] and model fit from eq. (1) for the steady-state creep rate of bulk Nb₅Si₃ and (Nb). The constants given in Table I were used for the calculations.

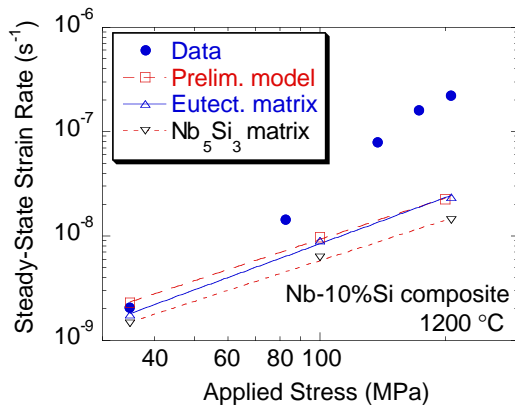
Figure 2. Comparison of the data [5] (open symbols) and model fit from eq. (1) (solid lines) for primary creep in bulk (Nb).

NUMERICAL RESULTS AND DISCUSSION

The steady-state creep data of Subramanian *et al.* [5] for the Nb-10% Si composite deformed at 1200 °C are compared with the FE predictions in Fig. 3 (the analytical model gives virtually the same results [7]). Model predictions are given for composites with a pure Nb₅Si₃ matrix and with the eutectoid matrix. The results of the preliminary study [7] are also given for comparison. The variation in the predicted strain rates among the models is small, being less

than a factor of two for applied stresses from 35 to 207 MPa. Thus, the details of modeling the microstructure do not significantly affect the FE predictions of steady-state creep in Nb₅Si₃/Nb *in-situ* composites. This finding is consistent with earlier calculations [7] showing that the predicted steady-state composite creep rate is insensitive to the properties of the discontinuous weak phase when the continuous strong matrix has a low stress exponent. Also note from Figs. 1 and 3 that the composite, which is about 73% (Nb), has a steady-state creep rate much lower than expected from a rule of mixtures. For example, at 90 MPa the composite creep rate is about a factor of 300 less than pure (Nb). This aspect of the data is qualitatively predicted by the models. Therefore, both the data and the models suggest that DPT composites such as Nb₅Si₃/Nb may have attractive creep properties.

(3)



(4)

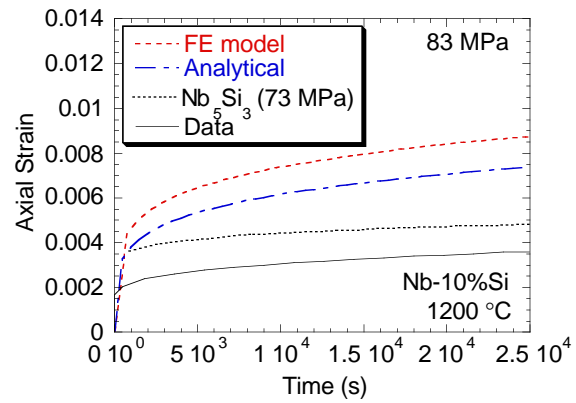


Figure 3. FE predictions of the steady-state creep rate as a function of applied stress for the Nb-10% Si composite are compared with the data of Subramanian *et al.* [5].

Figure 4. Primary creep curves computed by the two models for the Nb-10% Si composite deformed at an applied stress of 83 MPa are compared with the data of Subramanian *et al.* [5]. The data of Subramanian *et al.* [5] for Nb₅Si₃ deformed at 73 MPa are shown for reference.

Figure 3 shows that the continuum models predict a composite stress exponent nearly equal to that of the matrix, *i.e.* near unity. The data, however, show a much larger stress exponent of approximately 2.7. Thus, the steady-state creep rate predictions, which are accurate at low stress, are not accurate at high stresses. The failure of the FE (and analytical) models to correctly predict the composite stress exponent may be due to a breakdown of the continuum assumption in these microstructurally fine *in-situ* composites [8,15]. Since the major goal of this study was to assess the usefulness of the continuum approach to modeling creep in DPT composites, this possibility requires further investigation. Another potential reason for the underpredicted composite stress exponent is that the morphological details of the Nb₅Si₃ in the eutectoid matrix, which have not been modeled accurately, are important in determining the composite creep behavior. This possibility also is under investigation.

Figure 4 presents predictions of the FE and analytical models for primary creep in a composite with an Nb_5Si_3 matrix deformed at 83 MPa. The relatively minor difference in the two calculated creep curves stems from the more accurate representation by the FE model of the stress redistributions occurring during primary creep. The continuum models overpredict the primary creep strain and strain rate by about a factor of two. (At this stress the steady-state creep rate is *underpredicted* by about a factor of three, Fig. 3). While more accurate predictions are desirable, the errors may largely be due to the difficulties in experimentally measuring the primary creep curves. Notice that the 73 MPa pure Nb_5Si_3 curve in Fig. 4 lies *above* that of the composite deformed at 83 MPa, demonstrating the difficulty in performing these experiments in which the creep rates and strains are low. Since the 73 MPa Nb_5Si_3 data were used to evaluate the matrix material constants in the models, the predicted composite creep strains are necessarily above those of the data. Given these experimental uncertainties, the predicted primary creep curves are not unreasonable. As more data become available, presumably more accurate material constants could be obtained and the accuracy of the predictions could be improved. Thus, in the case of primary creep the continuum approach appears to be promising, though calculations over a range of applied stresses are required to make a complete assessment.

In addition to predicting creep strains or strain rates, the FE models can be used to give qualitative insights regarding potential sites for creep damage [8-10]. Subramanian *et al.* [5] observed cracking normal to the loading direction in the Nb_5Si_3 phase of the eutectoid matrix during tertiary creep. Figure 5 shows that the FE model predicts large axial tensile stresses (as well as large principal and hydrostatic tensile stresses) in the region between adjacent elongated primary (Nb) dendrites, rather than at their ends. Consistent with these calculations, the cracks observed during tertiary creep typically were found in this region.

The computed stress contours in the matrix also suggest possible reasons why the steady-state creep rates are underpredicted by the models at high stresses, Fig. 3. First, although matrix cracking has not yet been observed during steady-state creep, this investigation is continuing, particularly at the high applied stresses. If present during steady state, matrix cracking would contribute to higher than predicted creep rates. The second possibility concerns a change in stress exponent for Nb_5Si_3 at high stress. Data for pure Nb_5Si_3 at 1400 °C show a change from $n = 1$ to $n = 3$ as the applied stress increases above 200 MPa [5]. For the composite deformed at 1200 °C and 207 MPa, calculations similar to those shown in Fig. 5 reveal that the von Mises effective stress in the matrix ranges from 430 to 640 MPa. These values are well above the stresses experimentally tested, Fig. 1. If at 1200 °C the silicide exhibits a change in n at high stresses similar to that observed at 1400 °C, then creep in the Nb_5Si_3 phase of the composite may actually be occurring in an $n = 3$ regime for modest applied stresses. The high stress creep behavior of the Nb_5Si_3 at 1200 °C is, therefore, under experimental investigation to test this hypothesis.

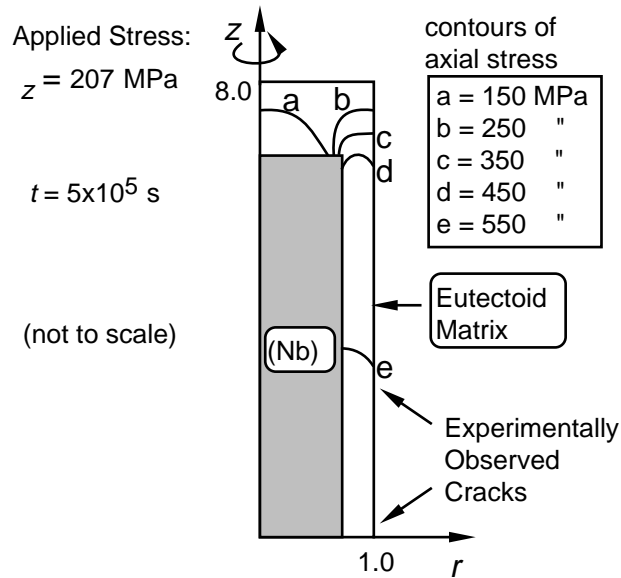


Figure 5. Contours of axial stress in the eutectoid matrix predicted by the FE model are shown following 5×10^5 s of deformation at an applied stress of 207 MPa.

SUMMARY AND CONCLUSIONS

Analytical and finite element continuum methods have been employed to model the primary and steady-state creep behavior of $\text{Nb}_5\text{Si}_3/\text{Nb}$ *in-situ* composites. These models incorporate a simple creep constitutive law that is capable of describing either transient or steady-state creep. The material constants in this equation have been estimated from experimental data for the bulk phases present in the composites. Creep damage effects were not included in the models. Based on calculations for a Nb-10 at.% Si composite deformed at 1200 °C, the following conclusions were reached.

1. Since the strong Nb_5Si_3 continuous phase has a low stress exponent ($n \approx 1.0$), the details of the microstructure (phase morphology and distribution) are not predicted to significantly affect the composite creep behavior. This prediction has yet to be experimentally verified.
2. While accurate at low stress, the models underpredict the steady-state composite creep rate at large stresses because the composite stress exponent is underpredicted.
3. At a stress of 83 MPa, the primary creep strains in the composite are overpredicted by about a factor of two. Given the uncertainties in the primary creep data for the composite and the bulk component phases, the predictions are reasonably accurate.
4. Finite element predictions of large tensile stresses in the matrix between adjacent elongated (Nb) dendrites are consistent with the experimentally observed location of cracks during tertiary creep.
5. Additional efforts are required to make firm conclusions regarding the viability of continuum models to predict the creep behavior of ductile-phase toughened composites.

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REFERENCES

- 1) M. G. Mendiratta, J. J. Lewandowski and D. M. Dimiduk, *Metall. Trans.* **22A**, 1573 (1991).
- 2) M. Thirukkonda, B. Cockeram, M. Saqib, L. E. Matson, R. Srinivasan and I. Weiss, *Scripta Metall. Mater.* **27**, 711 (1992).
- 3) B. P. Bewlay *et al.*, in *Processing and Fabrication of Advanced Materials III*, V. Ravi *et al.* eds., TMS, Warrendale, PA, pp. 547-565 (1994).
- 4) P. R. Subramanian *et al.*, in *High-Temperature Silicides and Refractory Alloys*, C. L. Briant *et al.* eds., Materials Research Society, Pittsburgh, PA, pp. 491-502 (1994).
- 5) P. R. Subramanian, T. A. Parthasarathy, and M. G. Mendiratta, presented at the 1994 MRS symposium on *High Temperature Ordered Intermetallic Alloys – VI*, Nov. 30, 1994, Boston, MA.
- 6) G. A. Henshall and M. J. Strum, *Scripta Metall. Mater.* **30**, 845 (1994).
- 7) G. A. Henshall and M. J. Strum, in *Intermetallic Matrix Composites III*, J. A. Graves, J. J. Lewandowski, and R. Bowman, eds., Materials Research Society, Pittsburgh, PA, pp. 65-70 (1994).
- 8) T. L. Dragone and W. D. Nix, *Acta Metall.* **38**, 1941 (1990).
- 9) T. Christman, A. Needleman, S. Suresh, *Acta Metall.* **37**, 3029 (1989).
- 10) V. Tvergaard, *Acta Metall. Mater.* **38**, 185 (1990).
- 11) B. E. Engelmann, "NIKE2D User Manual," University of California - Lawrence Livermore National Laboratory, UCRL-MA-105413 (1991).
- 12) R. G. Whirley and G. A. Henshall, *Int. J. Numer. Methods Engng.* **35**, 1427 (1992).
- 13) E. Bullock, M. McLean, and D. E. Miles, *Acta Metall.* **25**, 333 (1977).
- 14) M. Tanaka, T. Sakaki, and H. Iizuka, *Acta Metall. Mater.* **39**, 1549 (1991).
- 15) M. F. Bartholomeusz, Q. Yang, and J. A. Wert, *Scripta Metall. Mater.* **29**, 389 (1993).

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